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# COLLECTION AND ANALYSIS OF PARTICLES

FROM THE MESOPAUSE

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Collection and Analysis of Particles  
from the Mesopause\*

G. Witt, C. L. Hemenway and R. K. Soberman

ABSTRACT

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Particle sampling experiments utilizing sounding rockets were conducted in northern Sweden during the summer of 1962. Two successful flights were achieved, one (Aug. 11, 0240 local time) in the presence of noctilucent clouds and one (Aug. 7, 0147 local time) when no such clouds could be visually observed from the ground or from aircraft. The rockets were identically instrumented and insofar as can be determined, the experimental conditions of the two successful flights were similar. The collecting surfaces were exposed between the altitudes of approximately 75 and 95 kilometers during ascent only.

The collection surfaces from the cloud flight show a particle count of 4 to  $30 \times 10^{10} \text{ m}^{-2}$  (for  $d > 0.05$  micron) which is two to three orders of magnitude greater than that of the non-cloud flight or the controls. The size distribution is of the form  $N \propto d^{-p}$  where  $p$  ranges from 3 to 4. Evidence for Fe and Ni has been found in connection with the cloud particles. The larger particles show indications of having been surrounded by an ice coating.

Author

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Experiments for the collection of particles from the mesopause region of the atmosphere were conducted in northern Sweden during the summer of 1962.<sup>1,2</sup> The purpose of these experiments was to yield information about the nature of the particles of which noctilucent clouds are composed. The sampling devices were carried by Nike-Cajun sounding rockets fired from the Kronogård range located at latitude 66° N and longitude 19° E. The first rocket was launched on 7 August at 0147 local time in the absence of visible noctilucent clouds. Ground based and airborne observers were utilized to establish the presence or absence of the clouds. The second firing followed on 11 August at 0240 local time, in the presence of a display of noctilucent clouds which extended over the range. Figure 1 is a photograph taken of that display. The picture was taken by a ballistic camera located at an observational site in Kristineberg, about 150 kilometers south of the range. The camera was photographing the portion of the sky over the range. This display was also reported by other observers whose positions are indicated on the map in Figure 2. It was later established that a noctilucent cloud display was observed the following night over western Alaska.<sup>3</sup> These two successful flights were followed by two other attempts which failed due to malfunction of the payload separation system. Insofar as can be determined the flight conditions through separation with regard to trajectory and attitude were similar in all cases. The rockets reached an apogee of approximately 115 kilometers.

The experimental apparatus consisted of sampling surfaces mounted in cylindrical containers (See Figure 3). In each container were four high purity collection surfaces, (1) Nitrocellulose film with an evaporated coating of aluminum; (2) Nitrocellulose film coated with fuchsine dye; (3) Indium film and (4) Calcium film with a thin protective coating of aluminum, paraffin and silicone oil. The different surfaces were designed to retain particles or portions of particles that were non-volatile as well as indicate the presence of possible volatile particles or components thereof. To minimize contamination from terrestrial particles and to aid in the identification of such contaminants under electron microscopic examination, techniques similar to those employed in previous rocket samplings were used.<sup>4</sup> The primary controls consisted of areas shielded from particles moving in ballistic trajectories by the plastic hold-down plate visible in Figure 3. Between the plate and the control areas there was a 0.5 mm air gap which permitted contaminant particles (suspended in the air at ground level densities) to reach these areas, thus indicating the abundance and appearance of such contaminants.

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In each experiment the collection surfaces were exposed at an altitude of approximately 75 kilometers by spring ejection of an outer rocket nose tip. Figure 4 shows the configuration of the forward end of the payload where the two sampling containers were located. The two halves of the outer tip are also shown. A second spring mechanism closed the sampling ports and at the same time covered and vacuum sealed the sampling containers. This latter action occurred at an altitude of approximately 95 kilometers during the ascending portion of the flights. No attempt was made to sample during descent. The containers from the two successful flights were recovered sealed and in excellent condition within an hour after the launchings.

After the flights, the gas in the containers was examined by a mass spectrometer to determine if any volatile products foreign to the lower atmosphere had been obtained. Outgassing and gradual leakage of the containers were expected to mask slight concentration changes of gases or vapors normally found in the sea level environment. Only slight traces of ethyl alcohol and acetone, both used in the nose cone cleaning operations were detected by this technique.

The collection surfaces from the two flights exhibit significant differences. The first and most apparent of these is that the nitrocellulose films from the cloud flight contain non-volatile particles, the number per unit area of which are greater by two to three orders of magnitude than what was found on the surfaces from the non-cloud flights, and the contamination level as determined from the control areas. Figure 5 shows the comparison of electron micrographs of the aluminum shadowed nitrocellulose surfaces from (a) the cloud experiment; (b) the non-cloud experiment; and (c) a control area. While 5(a) is a typical field from that surface, 5(b) and 5(c) are not in the sense that the fields were selected to show a particle. A typical field in these latter cases would show no particles at all. The lighter areas in this figure are shadows of the particles cast by a coating which is evaporated on the surface at a low angle. After preparation of the surfaces a coating of aluminum was applied and on return to the laboratory this was followed by a second coating of chromium. Thus, particles deposited between the two coatings show one shadow, while those deposited before the first coating show two shadows and those deposited after the second coating show none. Another feature seen in Figure 5(a) is a circular pattern which surrounds most of the larger particles obtained from this flight. In Figure 6 we have a higher magnification electron micrograph of one large particle surrounded by such a pattern. Evidence from the calcium surfaces and laboratory simulation experiments indicate that this pattern was caused by an ice coating surrounding the particles at the time



of deposition which subsequently evaporated. To date in examining several thousand particles from the non-cloud flight and from the control areas only two or three such circular patterns have been seen. Figure 6 also shows dark spherical inclusions in the particle which are opaque to 100 KV electrons. This feature is observed in a large number of particles. Upon heating by prolonged intensive electron bombardment it is found that these opaque inclusions are far more stable than other portions of the particles.

Partial composition studies have been carried out using electron beam microprobes. Scanning of the cloud-exposed surface of the hold-down plates gives evidence of particles containing both Fe and Ni. Unfortunately the sensitivity of the microprobe is such that only particles with a diameter of about 1 micron or larger can be analyzed. A microprobe record for one particle is shown in Figure 7. The nickel to iron ratio shown here is approximately 0.1. Only two cases of such high ratios have been found to date, although traces of Ni are associated with many of the particles that give Fe indications.

A number of the particles show electron diffraction patterns with consistent d-values which have yet to be identified. Figure 8 shows a typical particle alongside the diffraction pattern obtained from the particle. The pattern is that of a twin crystal with hexagonal symmetry. The d-values for this pattern are listed below:

4.46 A  
2.575  
2.225  
1.684  
1.485  
1.285  
1.235  
1.112  
etc.

3.84 A-Sporadic reflection not belonging to the pattern.

The solid ring seen in the diffraction pattern is the (1,1,0) reflection of chromium with which the surfaces were coated. Traces of the (1,1,1) ring of aluminum, also part of the surface coating, can be seen in the figure.

The number of particles per unit area and the size distribution which were determined by scanning electron micrographs of the nitrocellulose collecting surfaces, exhibit variations which depend upon the position of the sampling surfaces in the nose cone. This variability is most likely due to the attitude of the vehicle relative to the particle drift as it passed through the cloud layer. The vehicle was spinning slowly (about 0.2 revolutions per second) and this was apparently not sufficient to average out the results. For particles larger than 0.05 microns in diameter our results show  $4 \text{ to } 30 \times 10^{10}$  particles per square meter. The size of these particles follow a distribution function of the form  $N \propto d^{-p}$  where  $p$ , in general, varies between 3 and 4 depending upon the sample. Such a size distribution is shown in Figure 9. Note the departure from the general distribution function that occurs at about  $d = 0.05$  micron. This feature occurs on all of the size distributions although the particle diameter at which it occurs varies slightly. This cut-off is not due to a decrease of the collection efficiency of the nose cone with particle size. Theoretical and experimental determinations of the collection efficiency at normal incidence show that this should not drop off appreciably for particle diameters greater than 0.01 micron. The number of particles per unit area and the size distributions are in agreement with the observed light scattering properties of the clouds.<sup>5,6</sup>

In Figure 10 we have plotted the percentage of particles surrounded by the ring pattern as a function of the diameter of the particle itself. By comparison with Figure 9, it is seen that most of the larger particles have the ring pattern whereas few of the smallest particles have such patterns.

Statistical and composition analyses of the particles from both the cloud and the non-cloud flights are continuing. A more detailed report will be published in the near future. Plans are presently being formulated for similar experiments to measure parameters such as particle concentration and size as function of the altitude of collection. With refinement of the collector design it is hoped that better quantitative data can be obtained from future flights.

## ACKNOWLEDGMENTS

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## LEGENDS

1. Photograph of noctilucent cloud display over Kronogård, 11 August 1962.
2. Map of Scandinavia showing the locations of reported sightings of the cloud display of 11 August 1962.
3. Particle sampling container showing collecting surfaces and hold-down plate.
4. Forward section of collector payload with halves of the ejectable outer tip.
5. Electron micrograph of (a) cloud collecting surface, (b) non-cloud collecting surface and (c) control surface. (scale = 1 micron).
6. Electron micrograph of a noctilucent cloud particle with ring structure. (scale = 1 micron).
7. Electron beam microprobe record showing relative Ni/Fe ratio from a particle recovered from the cloud flight.
8. (a) Electron micrograph of a noctilucent cloud particle and (b) corresponding electron diffraction pattern. (scale = 1 micron).
9. Cloud particle size distribution from a nitrocellulose collecting surface.
10. Percentage of particles with ring patterns.



FIGURE 1

PHOTOGRAPH OF NOCTILUCENT CLOUD DISPLAY OVER  
KRONOGARD, 11 AUGUST 1962

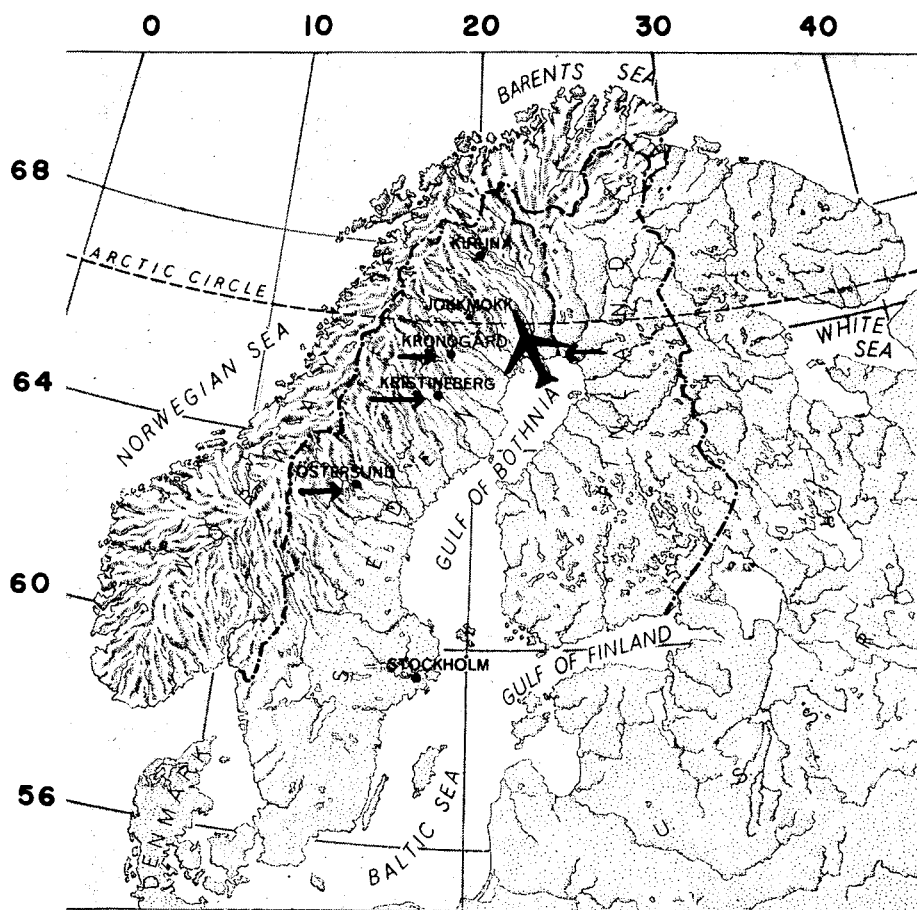


FIGURE 2

MAP OF SCANDINAVIA SHOWING THE LOCATIONS OF  
REPORTED SIGHTINGS OF THE CLOUD

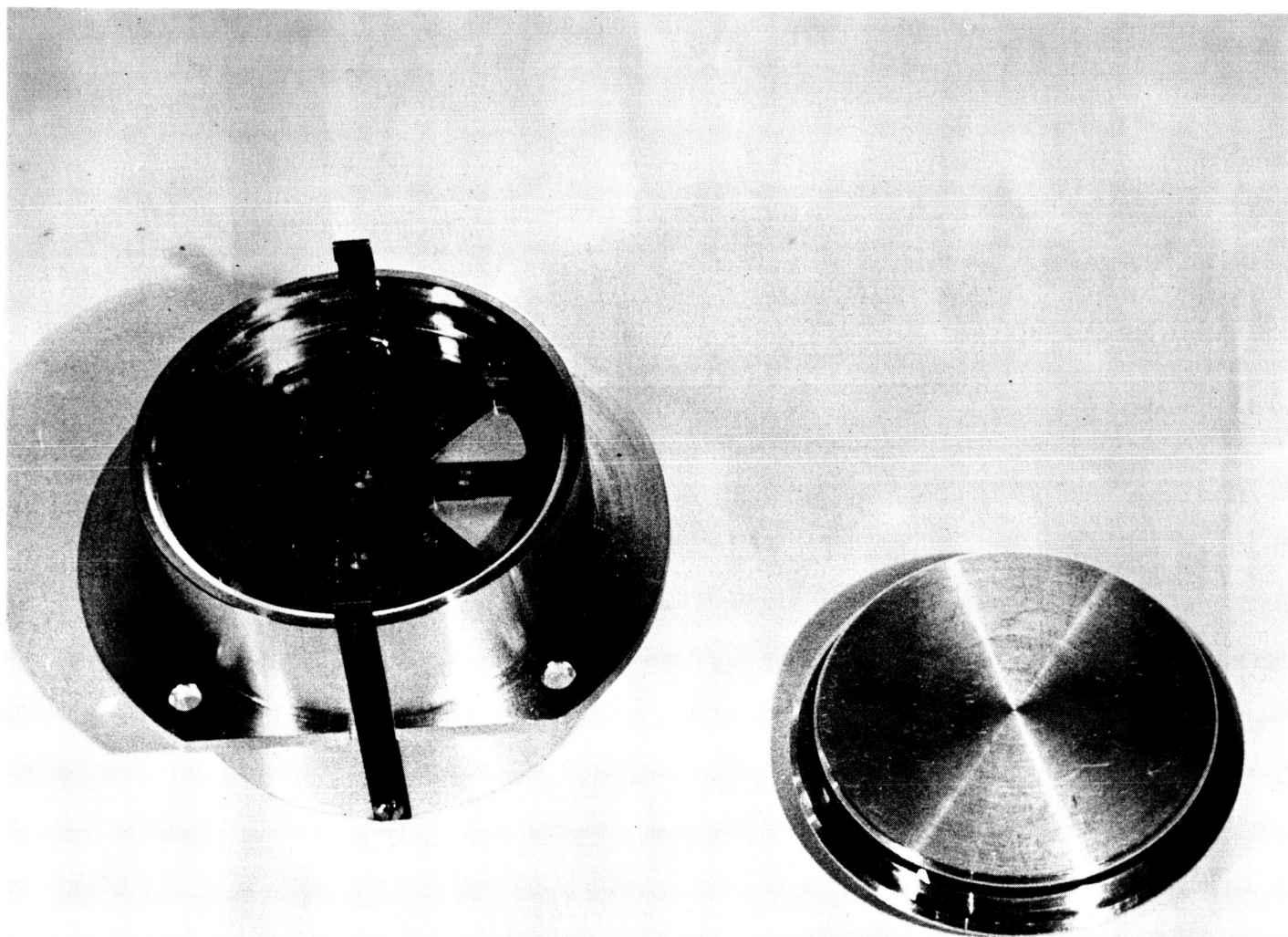


FIGURE 3

PARTICLE SAMPLING CONTAINER SHOWING COLLECTING  
SURFACES AND HOLD-DOWN PLATE



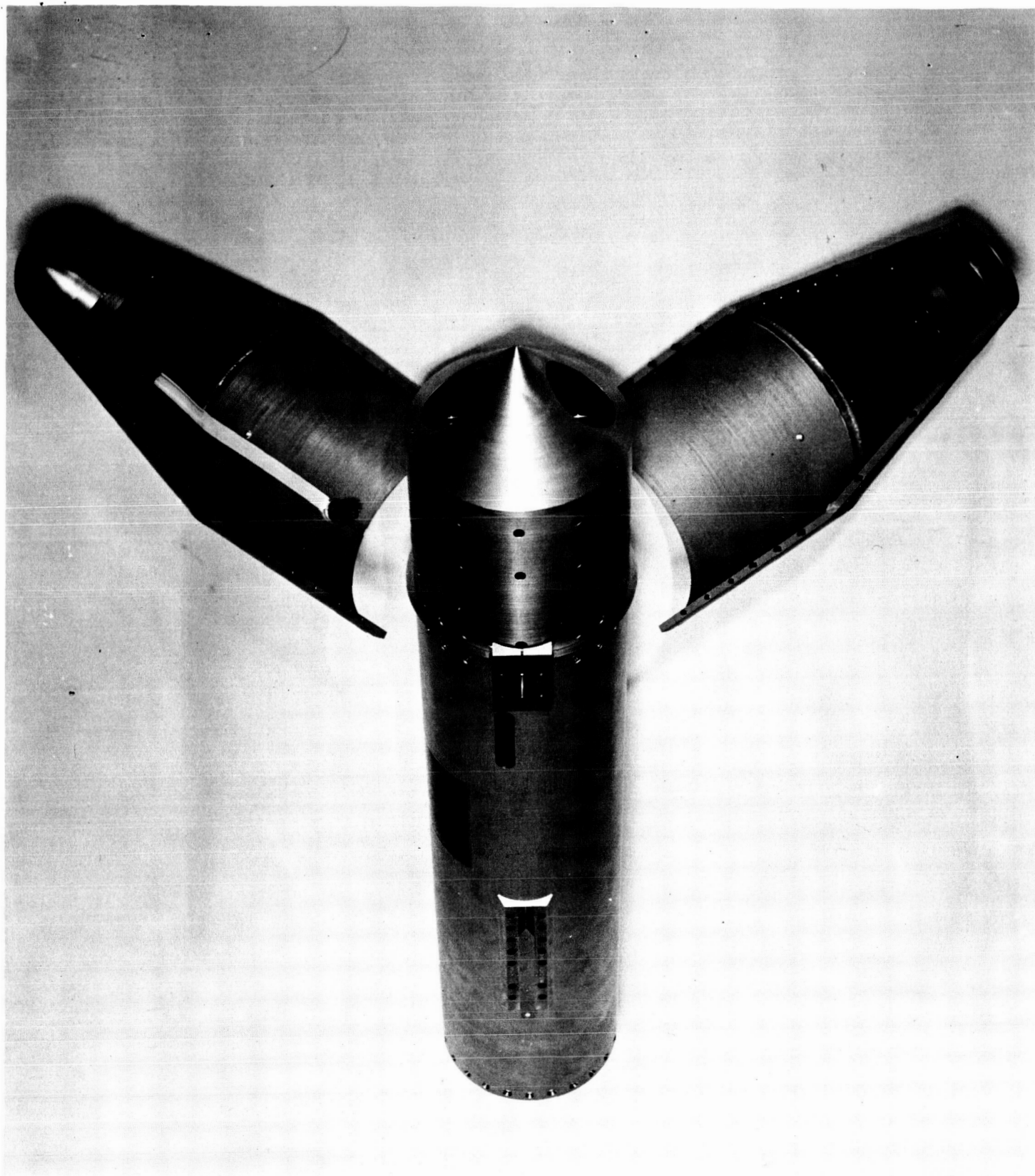
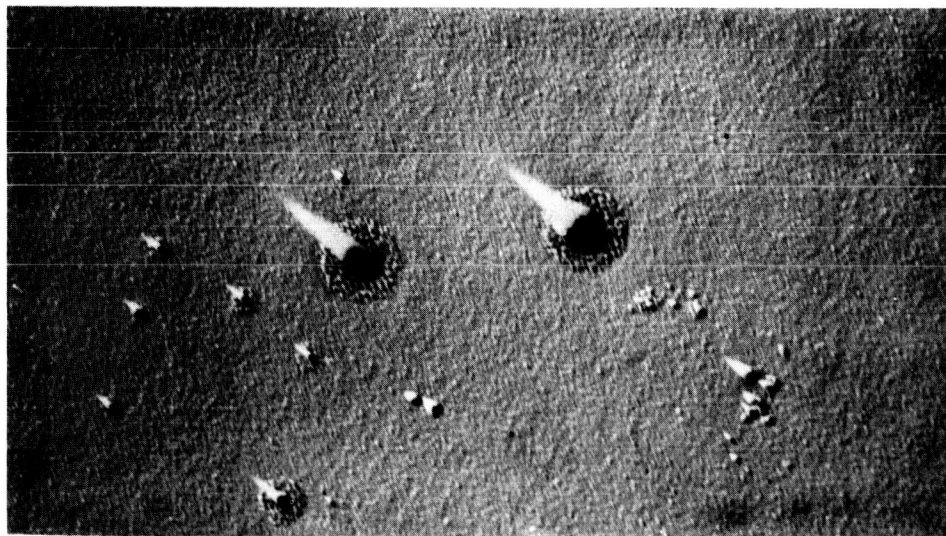
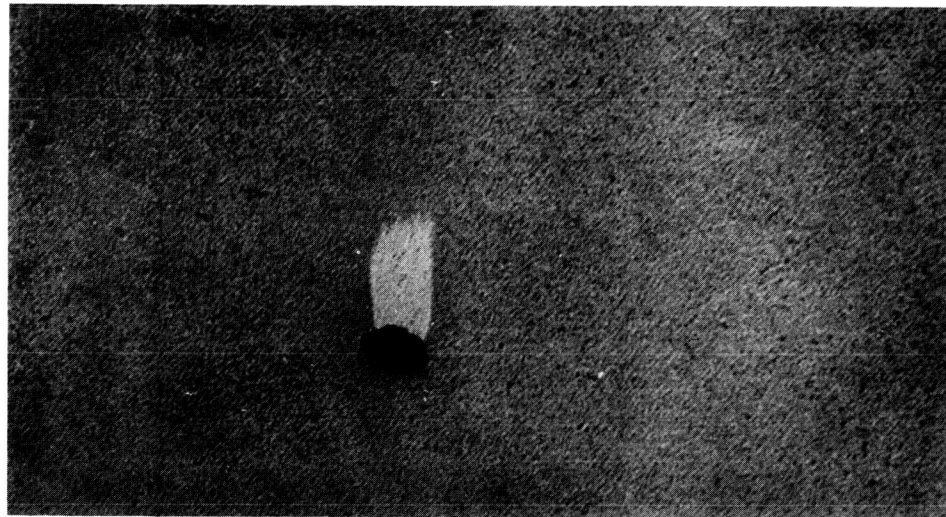


FIGURE 4

FORWARD SECTION OF COLLECTOR PAYLOAD WITH  
HALVES OF THE EJECTABLE OUTER TIP



A



B



C

FIGURE 5

ELECTRON MICROGRAPH OF (a) CLOUD COLLECTING  
SURFACE, (b) NON-CLOUD COLLECTING SURFACE AND (c) CONTROL SURFACE  
(SCALE = 1 MICRON)

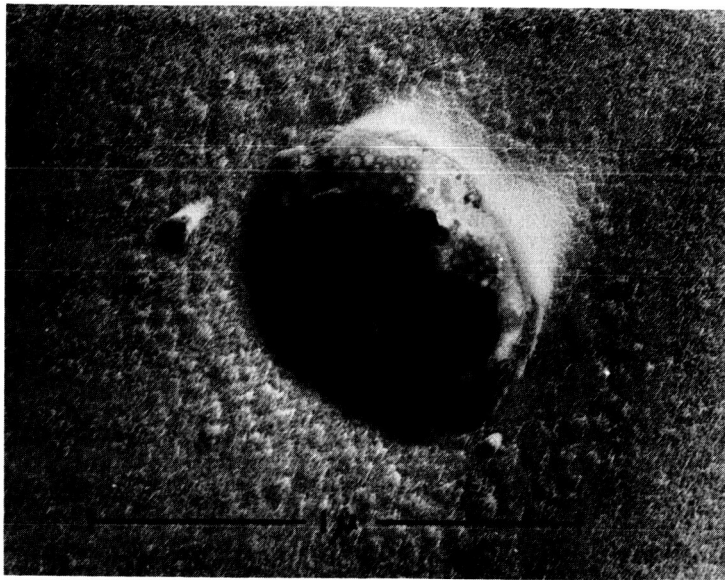
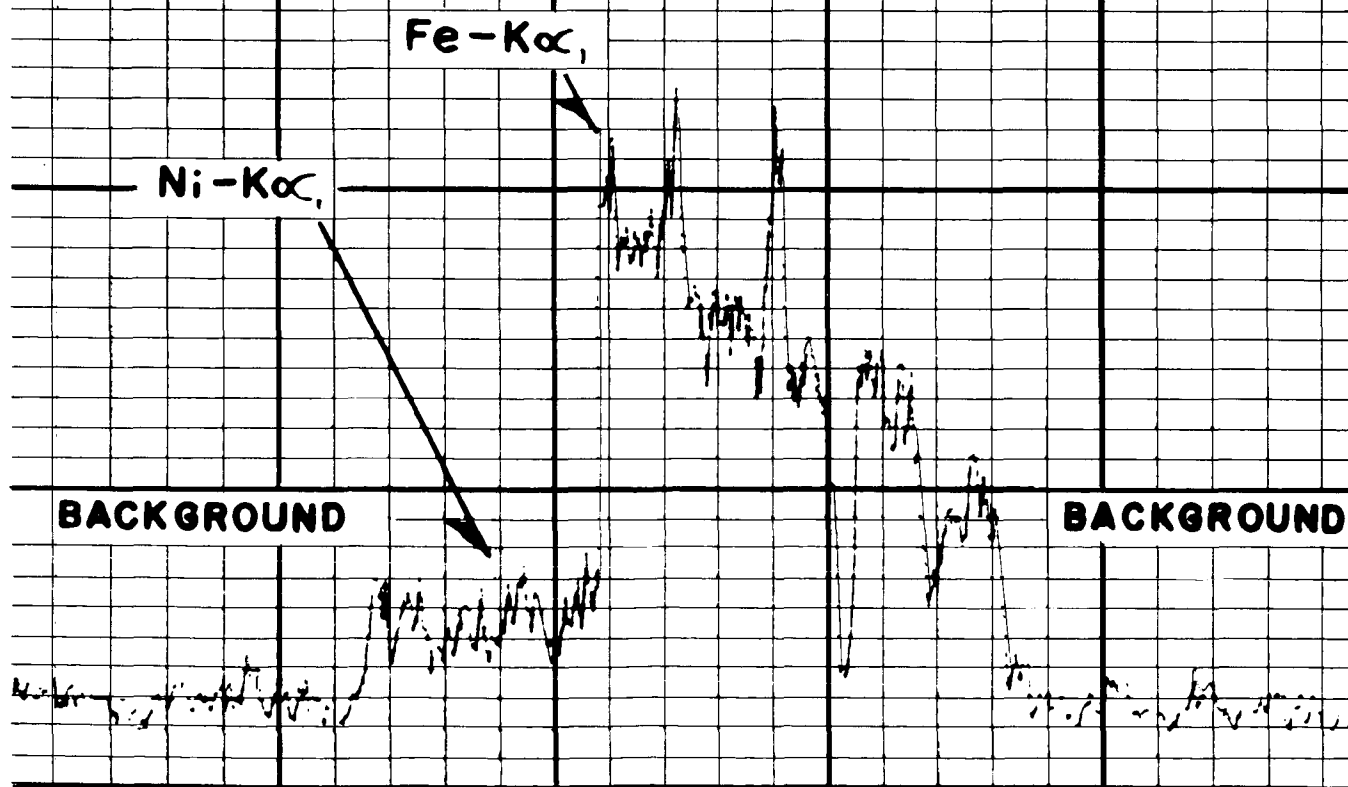


FIGURE 6

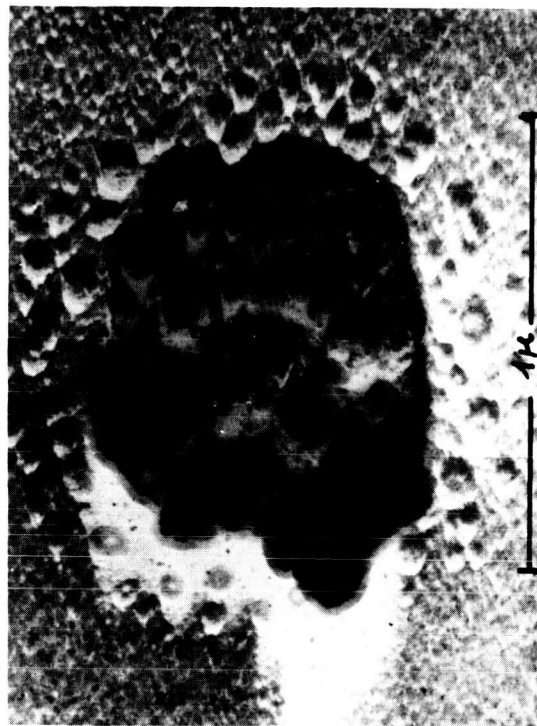
ELECTRON MICROGRAPH OF A NOCTILUCENT  
CLOUD PARTICLE WITH RING STRUCTURE  
(SCALE = 1 MICRON)

**ELECTRON BEAM MICROPROBE  
RECORD FOR PARTICLE FROM  
NATTPIP II (11 AUG 1962)**

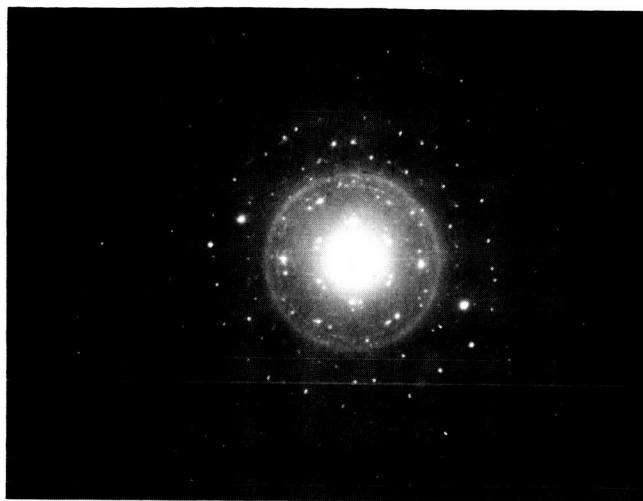


**FIGURE 7**

**ELECTRON BEAM MICROPROBE RECORD SHOWING RELATIVE Ni/Fe  
RATIO FROM A PARTICLE RECOVERED FROM THE CLOUD FLIGHT**



A



B

FIGURE 8

(a) ELECTRON MICROGRAPH OF A NOCTILUCOUS CLOUD PARTICLE  
AND (b) CORRESPONDING ELECTRON DIFFRACTION PATTERN  
(SCALE = 1 MICRON)

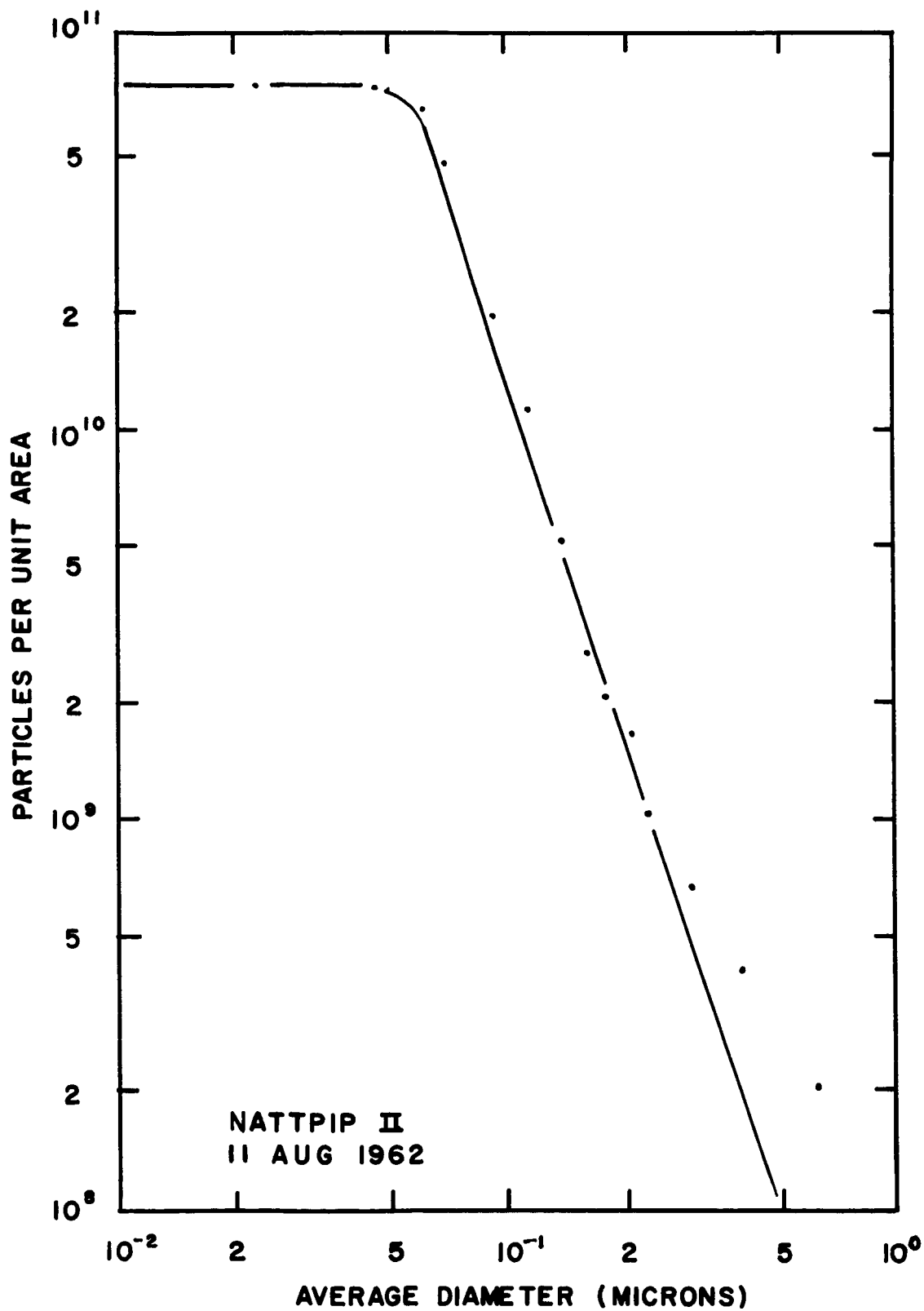


FIGURE 9

CLOUD PARTICLE SIZE DISTRIBUTION FROM A  
NITROCELLULOSE COLLECTING SURFACE

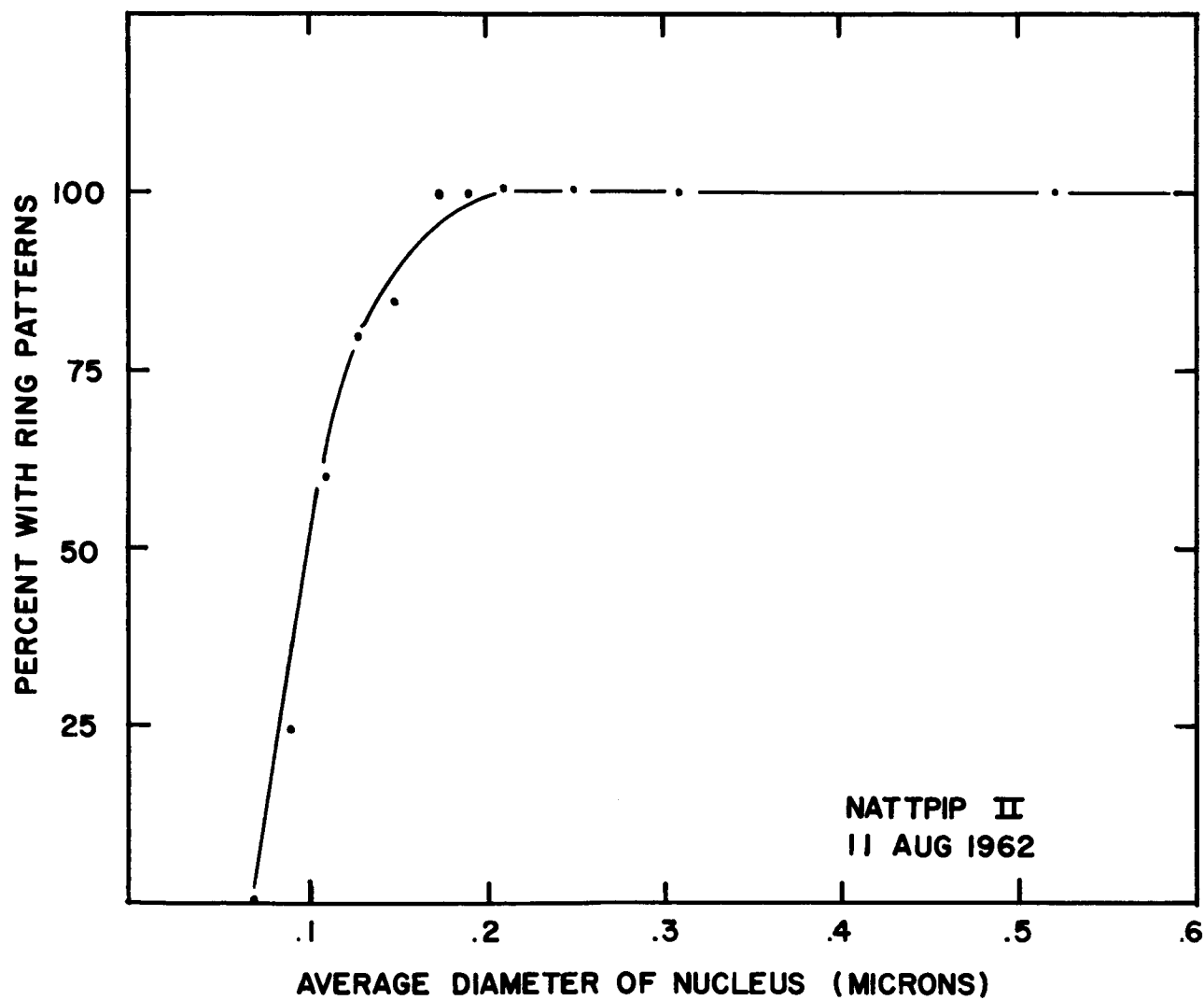


FIGURE 10

PERCENTAGE OF PARTICLES WITH RING PATTERNS